

# An Assessment of Winery Wastewater Diluted for Irrigation of Grapevines in the Breede River Valley with Respect to Water Quality and Nutrient Load

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Possible re-use of winery wastewater for irrigation was investigated in a field trial with micro-sprinkler-irrigated Cabernet Sauvignon/99 Richter in the Breede River Valley region of South Africa. Irrigation with winery wastewater diluted to 100, 250, 500, 1 000, 1 500, 2 000, 2 500 and 3 000 mg/L chemical oxygen demand (COD), respectively, was compared to irrigation with raw river water. Since the pH was lower than 6, the diluted wastewater could cause nutrient toxicity. The diluted winery wastewater did not pose any salinity hazard, as the electrical conductivity was well below 2 dS/m. For the given range of dilutions, the sodium adsorption ratio never exceeded 10, which indicates that the water posed no sodicity hazard. Sodium and  $\text{Cl}^-$  never exceeded 115 and 150 mg/L, the respective upper thresholds for grapevines. With the exception of N, levels of  $\text{H}_2\text{PO}_4^-$ ,  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{HCO}_3^-$ ,  $\text{SO}_4^{2-}$  and  $\text{B}^{3+}$  in the diluted wastewater increased with a decrease in dilution level. The N load in diluted winery wastewater appeared to be completely inadequate to supply the grapevine's requirements. In contrast, the P load in the winery wastewater diluted to 2 500 mg/L COD and higher would supply more than adequate P if the grape yield amounts to 10 t/ha. Likewise, the dilution of winery wastewater to 250 mg/L COD and higher would supply more than adequate  $\text{K}^+$  if the grape yield amounts to 10 t/ha. However,  $\text{K}^+$  applied *via* the wastewater will only be beneficial if it is not leached from the root zone during winter.

## INTRODUCTION

Wineries produce large volumes of low-quality wastewater, particularly during the harvest period. Reports on the actual volumes of wastewater generated by wineries are extremely limited. However, it is estimated that medium to large wineries generate more than 15 000 m<sup>3</sup> of wastewater annually, whereas small wineries generate less than 15 000 m<sup>3</sup> (Van Schoor, 2005 and references therein). Australian wineries generate about 3 to 5 m<sup>3</sup> of wastewater per ton of grapes crushed, with high organic load, variable salinity and nutrient levels (Chapman *et al.*, 1995; Mosse *et al.*, 2011). Crushing approximately 50 000 tons of grapes annually generates about 175 000 m<sup>3</sup> of wastewater at the Berri Estates Winery in the Riverland region of South Australia (Anonymous, 2010). Hence, their wastewater generation amounts to c. 3.5 m<sup>3</sup> per ton of grapes. It can be estimated that the winery at Lutzville Vineyards generates about 1.1 m<sup>3</sup> of wastewater per ton of grapes crushed. However,

this relatively low value is misleading, since 50% of the wastewater is presumably lost to evaporation (Kriel, 2008).

Winery wastewater contains high levels of  $\text{K}^+$  and  $\text{Na}^+$  (Laurenson *et al.*, 2012). Although various parameters may be used to evaluate winery wastewater, chemical oxygen demand (COD), pH, sodium adsorption ratio (SAR), electrical conductivity of the irrigation water ( $\text{EC}_{\text{iw}}$ ),  $\text{Cl}^-$ ,  $\text{K}^+$  and  $\text{Na}^+$  are considered to be important. A survey carried out to evaluate winery wastewater generated by the South African wine industry revealed that the water quality parameters vary substantially between wineries (Mulidzi *et al.*, 2009). The variation in water quality parameters also occurs in wastewater produced by wineries all over the world (Conradie *et al.*, 2014 and references therein). Furthermore, a strong seasonal variation in winery wastewater quality was observed in the South African industry (Mulidzi *et al.*, 2009). A similar seasonal trend was reported for winery wastewater in Australia (Arienzo *et al.*, 2009a). These trends were

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confirmed where effluents of two wineries were monitored frequently (Sheridan *et al.*, 2011). Considering the legal requirements for irrigation water quality in South Africa, the results of the survey confirmed that the majority of South African wineries are not able to irrigate crops beneficially as part of the General Authorisations for irrigation with winery wastewater, unless the water is first subjected to an effective form of pre-treatment or unless there is relaxation of the General Authorisations (Department of Water Affairs & Forestry, 1996; Department of Water Affairs, 2013).

International requirements, as well as national legislation, are putting pressure on wine producers regarding the responsible management of their wastewater, which may have a large-scale detrimental impact on the environment. Most vineyards in the Western Cape need irrigation. Therefore, the ideal situation would be to implement the sustainable use of winery wastewater for wine grape irrigation by adding winery wastewater to existing irrigation water resources. This practice has already been performed by various wineries for decades. Until now, however, the impact of this practice has not been studied comprehensively. Currently, the Department of Water and Sanitation is drafting a new Authorisation for wineries. Depending on the permitted water quality limits and volumes stipulated by these authorisations, adding winery wastewater to current irrigation water may well become a more viable practice in the future. Re-using winery wastewater in this way will be beneficial, particularly where water shortages occur.

The objectives of this study were to determine whether winery wastewater quality could be improved by dilution with raw river water for irrigation, and to quantify the amount of plant nutrients applied *via* irrigation with the diluted water.

## MATERIALS AND METHODS

### Experiment vineyard

The re-use of winery wastewater for irrigation was investigated in a field trial with micro-sprinkler-irrigated Cabernet Sauvignon/99 Richter in the Breede River Valley region of South Africa. The locality is at 33° 41' latitude and has a Mediterranean climate. Based on the growing degree days (GDD) from September until March (Winkler, 1962),

the specific locality is in a class V climatic region for wine quality potential (Le Roux, 1974). The vineyard is located on an alluvial flood plain of the Du Toitskloof Mountains. The sandy soil belongs to the Longlands form (Soil Classification Working Group, 1991). The soil was deep delved to 1.0 m before planting. Grapevines were planted 2.4 m × 1.2 m and trained onto a four-strand lengthened Perold trellis (Booyesen *et al.*, 1992). Vertical shoot positioning was carried out to prevent the development of a sprawling canopy.

### Experiment layout

Irrigation with winery wastewater diluted to 100, 250, 500, 1 000, 1 500, 2 000, 2 500 and 3 000 mg/L COD with river water was compared to a control irrigated with river water (Table 1). All treatments were replicated three times in a randomised block design. Experiment plots comprised two rows of six grapevines each, with two buffer grapevines at each end and a buffer row on each side. Each experiment plot covered 104 m<sup>2</sup>. Treatments were applied from 2009/2010 until 2012/2013.

### Irrigation volumes applied

Details of the irrigation infrastructure, as well as the dilution procedures, have been reported elsewhere (Myburgh *et al.*, 2015). Due to the delayed completion of the infrastructure installation in the 2009/2010 season, the application of the diluted wastewater treatments was only possible after harvest. In the following years, the wastewater irrigation treatments were applied from mid-February, when high volumes of wastewater usually become available as the harvest period begins in the Breede River valley. After each wastewater irrigation application, grapevines were also irrigated with river water to flush the irrigation pipes. Grapevines were generally irrigated at *c.* 50% plant available water (PAW) depletion to prevent excessive vegetative growth and yield reduction. Irrigation had to be applied every two weeks to maintain this PAW depletion level. Irrigation was terminated either in mid-April or the beginning of May each year, when the wastewater volumes decreased and the first winter rains began. Water meters were used to monitor the irrigation volumes applied to each treatment. Grapevines of all treatments, including those of the control, received the same volume of water per irrigation.

TABLE 1

Total amount of irrigation water applied per season to Cabernet Sauvignon/99R grapevines during the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	Amount per season (mm)			
	2009/2010	2010/2011	2011/2012	2012/2013
T1 - River water	178.5	391.6	246.9	294.7
T2 - 100	162.5	395.9	247.0	294.7
T3 - 250	163.2	402.8	246.9	297.0
T4 - 500	164.8	399.6	246.8	294.0
T5 - 1 000	167.5	399.2	246.9	293.3
T6 - 1 500	166.8	405.7	247.2	297.6
T7 - 2 000	161.1	398.8	246.9	293.2
T8 - 2 500	162.0	393.7	246.9	289.8
T9 - 3 000	159.5	395.3	246.8	294.1

### Water quality

Approximately one hour after a wastewater irrigation started, a 500 mL water sample was collected from each of the eight dilution tanks to verify actual COD levels obtained for each of the eight dilution treatments. A 500 mL sample of the river water was collected at the same time. Samples of the undiluted winery wastewater from the stock dam and river, as well as the samples from the eight dilution tanks, were analysed by a commercial laboratory for pH,  $EC_{iw}$ ,  $NH_4^+-N$ ,  $NO_3^--N$ ,  $H_2PO_4^-$ ,  $Ca^{2+}$ ,  $Mg^{2+}$ ,  $K^+$ ,  $Na^+$ ,  $Cl^-$ ,  $HCO_3^-$ ,  $SO_4^{2-}$ ,  $B^{3+}$ ,  $Fe^{2+}$  and heavy metals according to methods described by Clesceri *et al.* (1998). The COD in samples was measured using a portable spectrophotometer (Aqualitic COD-reactor®, Dortmund) with the appropriate test kits (COD, CSB, 0-15 000 mg/L). The potassium adsorption ratio (PAR) was calculated as follows:

$$PAR = K^+ \div [(Ca^{2+} + Mg^{2+}) \div 2]^{0.5} \quad (\text{Eq. 1})$$

where  $K^+$  is the potassium concentration (mg/L) divided by the molecular mass, *i.e.* 39 g/mol,  $Ca^{2+}$  is the calcium concentration (mg/L) divided by the equivalent molecular mass, *i.e.* 20 g/mol, and  $Mg^{2+}$  is the magnesium concentration (mg/L) divided by the equivalent molecular mass, *i.e.* 12.15 g/mol. Similarly, the sodium adsorption ratio (SAR) was calculated as follows:

$$SAR = Na^+ \div [(Ca^{2+} + Mg^{2+}) \div 2]^{0.5} \quad (\text{Eq. 2})$$

where  $Na^+$  is the sodium concentration (mg/L) divided by the molecular mass, *i.e.* 23 g/mol. The  $NH_4^+-N$  and  $NO_3^--N$  concentrations were summed to obtain the total N. No N determinations were carried out in the 2009/2010 season. The assessment of the microbial status of the winery wastewater, as well the diluted waters, was beyond the scope of the study.

### Amount of elements applied

For the diluted wastewater treatments, *i.e.* T2 to T9, the amount of wastewater applied was converted from mm to L per ha as follows:

$$V = I \times 10^4 \quad (\text{Eq. 3})$$

where  $I$  is the amount of irrigation applied (mm) and  $10^4$  is the factor used to convert depth of water (mm) to volume (L) per hectare ( $1 \text{ mm} = 10 \text{ m}^3 \text{ per ha} = 10^4 \text{ L per ha}$ ).

For each treatment, the element concentrations in the diluted wastewater were used to calculate the amounts of elements added to the soil per irrigation per hectare, as follows:

$$m = V \times C_e \quad (\text{Eq. 4})$$

where  $m$  is amount of the element (mg/ha),  $V$  is the volume of water applied per hectare (L) and  $C_e$  is the element concentration (mg/L) in the irrigation water.

In addition, the contribution of the elements deposited by the river water was taken into account. The same procedure was followed for T1, *i.e.* the river water control treatment. The amount of element in milligram per hectare was converted to kilogram per hectare (M), as follows:

$$M = m \div 1000 \div 1000 \quad (\text{Eq. 5})$$

The amount of elements applied per irrigation were summed to obtain the seasonal application.

### Statistical analysis

Mean values and standard deviations were calculated for pH,  $EC_{iw}$ ,  $H_2PO_4^-$ ,  $Ca^{2+}$ ,  $Mg^{2+}$ ,  $K^+$ ,  $Na^+$  and SAR for each season, namely 2009/2010, 2010/2011, 2011/2012 and 2012/2013. In the case of total N, means and standard deviations were calculated for the 2010/2011, 2011/2012 and 2012/2013 seasons. For  $Cl^-$ ,  $HCO_3^-$ ,  $SO_4^{2-}$ ,  $B^{3+}$  and  $Fe^{2+}$ , means and standard deviations were calculated as the average over the four seasons.

## RESULTS AND DISCUSSION

### Irrigation volumes applied

A full tank of diluted winery wastewater applied *c.* 41 mm irrigation to the three replication plots of each treatment. Grapevines of all treatments received the same mean volume of wastewater per irrigation when a full tank of wastewater was applied to the plots. Due to low COD levels in the winery wastewater in the middle of April in the 2010/2011 season, T3 to T9 had to be applied in two irrigations on consecutive days to apply the required COD. The COD applied on these consecutive days were summed. Due to low COD levels in the winery wastewater, as well as limited available water, only 1/3 of a tank of diluted water could be applied to each treatment on 2 May in the 2011/2012 season. This data was therefore not considered for the calculation of the average mean volume of wastewater applied per irrigation. Grapevines of the diluted wastewater treatments received only *c.* 25 mm, 14 mm and 8 mm of river water per irrigation in 2010/2011, 2011/2012 and 2012/2013 respectively. The amount of river water applied following wastewater irrigations was considerably less in 2012/2013 than in previous years, since it was decided to follow a continuous deficit irrigation strategy to minimise drainage losses and to curtail possible excessive vegetative growth. The total seasonal irrigation water amounts applied are presented in Table 1.

### Water quality

During the four seasons, water pH tended to decrease with a decrease in the dilution of the wastewater, *i.e.* an increase in the COD level of the water (Table 2). The pH of the river water (T1) was generally the highest, whereas pH in winery wastewater diluted to COD levels of 1 500 mg/L and higher were comparable to the undiluted winery wastewater, *i.e.* T5 to T9. In general, water pH levels tended to be below the recommended pH for irrigation water, which ranges from 6.5 to 8.4 (Department of Water Affairs & Forestry, 1996). It should be noted that, even though the pH of the river water was higher than the diluted wastewater treatments, at times it was also below the recommended levels for irrigation. With regard to the General Authorisations of 2013, up to 500 m<sup>3</sup> of wastewater may be irrigated on any given day, provided that the pH is between 6 and 9 (Department of Water Affairs, 2013). Due to the low pH (Table 2), problems with the corrosion of metals and concrete used in irrigation systems and infrastructure can be expected when the irrigation water has a pH below 6.5 (Department of Water Affairs & Forestry,

TABLE 2

The pH and electrical conductivity ( $EC_{iw}$ ) in winery wastewater, river water and diluted winery wastewater used for irrigation of Cabernet Sauvignon/99R during the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	pH				$EC_{iw}$ (dS/m)			
	2009/2010	2010/2011	2011/2012	2012/2013	2009/2010	2010/2011	2011/2012	2012/2013
Winery	5.4 ± 0.3	4.3 ± 0.8	4.7 ± 0.3	4.4 ± 0.5	2.30 ± 0.93	1.50 ± 0.51	1.63 ± 0.39	1.11 ± 0.28
T1 - River water	6.6 ± 0.5	5.8 ± 0.5	6.1 ± 0.5	5.5 ± 0.5	0.13 ± 0.02	0.19 ± 0.02	0.13 ± 0.07	0.12 ± 0.06
T2 - 100	6.6 ± 0.2	5.7 ± 0.8	5.9 ± 0.2	5.6 ± 0.5	0.16 ± 0.02	0.20 ± 0.03	0.16 ± 0.07	0.13 ± 0.06
T3 - 250	6.4 ± 0.2	5.5 ± 0.9	5.3 ± 0.2	5.1 ± 0.5	0.19 ± 0.03	0.22 ± 0.04	0.19 ± 0.06	0.15 ± 0.06
T4 - 500	5.8 ± 0.5	4.9 ± 0.8	4.9 ± 0.5	4.7 ± 0.8	0.26 ± 0.04	0.27 ± 0.05	0.25 ± 0.03	0.19 ± 0.06
T5 - 1 000	5.5 ± 0.5	4.4 ± 0.6	4.8 ± 0.5	4.6 ± 0.8	0.42 ± 0.13	0.36 ± 0.08	0.34 ± 0.06	0.24 ± 0.07
T6 - 1 500	5.4 ± 0.7	4.6 ± 0.9	4.7 ± 0.7	4.5 ± 0.8	0.53 ± 0.20	0.43 ± 0.10	0.42 ± 0.11	0.27 ± 0.07
T7 - 2 000	5.3 ± 0.6	4.4 ± 0.9	4.6 ± 0.6	4.5 ± 0.8	0.64 ± 0.26	0.53 ± 0.17	0.50 ± 0.20	0.32 ± 0.09
T8 - 2 500	5.5 ± 0.4	4.4 ± 1.0	4.6 ± 0.4	4.4 ± 0.8	0.74 ± 0.29	0.59 ± 0.21	0.61 ± 0.25	0.37 ± 0.10
T9 - 3 000	5.4 ± 0.4	4.4 ± 0.9	4.6 ± 0.4	4.4 ± 0.8	0.87 ± 0.36	0.69 ± 0.30	0.66 ± 0.24	0.42 ± 0.11

1996). The diluted wastewater could also cause problems, such as  $Al^{3+}$ ,  $Mn^{2+}$  and heavy metals being mobilised to concentrations high enough to be toxic to grapevines if used for irrigation (Howell & Myburgh, 2013). The foregoing indicates that the dilution of winery wastewater did not have any positive effect on pH in terms of irrigation water quality, irrespective of the level of dilution.

The  $EC_{iw}$  increased with a decrease in the level of dilution of the wastewater (Table 2). This indicates that there was an increase in salt levels with a decrease in the dilution of the wastewater. Furthermore, the  $EC_{iw}$  in the least diluted water, *i.e.* wastewater diluted to a COD level of 3 000 mg/L, was still substantially lower compared to the undiluted winery wastewater. This trend was consistent for all four seasons. On 2010-04-12, the  $EC_{iw}$  in the wastewater diluted to 2 000 mg/L COD and higher, *i.e.* T7, T8 and T9 (data not shown), exceeded the critical value of 0.75 dS/m, which is the salinity threshold for water used for grapevine irrigation (Van Zyl, 1981; Myburgh, 2012). This salinity threshold was also exceeded in wastewater diluted to a COD level of 3 000 mg/L (T9) on 2010-05-03 (data not shown). The  $EC_{iw}$  in the T7, T8 and T9 waters, *i.e.* wastewater diluted to COD levels of 2 000 mg/L and higher, exceeded the critical value of 0.75 dS/m on 2011-04-14 and 2012-05-02 (data not shown). In 2012/2013, the  $EC_{iw}$  did not exceed the critical value of 0.75 dS/m. In general,  $EC_{iw}$  in the diluted winery wastewaters was well below 2 dS/m, *i.e.* the norm according to the General Authorisations of 2013 (Department of Water Affairs, 2013). Given that the EC in the undiluted water was close to 2 dS/m, the dilution of winery wastewater had a positive effect on the salinity hazard in terms of irrigation water quality.

With the exception of the first season, the mean COD levels in the diluted winery wastewater were generally close to the target values (data not shown). In general, the COD in the diluted winery wastewaters was well below the norms stipulated by the General Authorisations of 2013 (Department of Water Affairs, 2013). Given that the COD in the undiluted winery water was generally not lower than 7 624 mg/L, the dilution of winery wastewater had a positive effect on the COD in terms of irrigation water quality.

In general, there were no consistent trends with regard to  $NH_4^+-N$ ,  $NO_3^- -N$  (data not shown) and total N concentrations, which could be related to the level of dilution (Table 3). However, it was evident that the N levels in the river water were higher compared to that of some of the diluted wastewater treatments. At this stage there is no explanation for the latter trend. Levels of  $H_2PO_4^-$  in the diluted wastewater treatments increased with a decrease in level of dilution (Table 3). Although there are no guidelines for  $H_2PO_4^-$  levels in irrigation water, a long-term critical value of 0.05 mg/L has been recommended by ANZECC (2000). This norm has been established to minimise the risk of algal blooms developing in storage facilities and to reduce the likelihood of bio-fouling (biological fouling) in irrigation equipment. The levels in the diluted wastewaters, as well as undiluted winery wastewater, generally exceeded this norm (Table 3), thereby indicating that  $H_2PO_4^-$  in winery wastewater may induce algal blooms. Since the latter could cause problems in irrigation systems, the dilution of the winery wastewater did not seem to have had any positive effect in terms of  $H_2PO_4^-$  concentration.

The levels of  $Ca^{2+}$  increased with a decrease in the dilution of the winery wastewater (Table 4). There are no guidelines for  $Ca^{2+}$  levels in irrigation water (Department of Water Affairs & Forestry, 1996). However, it is important to determine the  $Ca^{2+}$  levels to calculate the SAR. As  $Ca^{2+}$  is beneficial, rather than harmful, to soil structure, it may ameliorate the impacts of Na application indirectly *via* its role in reducing the SAR. However, if the  $Ca^{2+}$ -to- $Mg^{2+}$  ratio in the water is less than one, the potential negative effects of  $Na^+$  may be exacerbated (Ayers & Westcott, 1985). Magnesium concentrations increased with a decrease in the dilution of the winery wastewater (Table 4), but there were no substantial differences in  $Mg^{2+}$  levels with regard to the dilution treatments. There are no guidelines for  $Mg^{2+}$  levels in irrigation water (Department of Water Affairs & Forestry, 1996). Similar to  $Ca^{2+}$ ,  $Mg^{2+}$  can also play an indirect, positive role in reducing the SAR. On the negative side, crops irrigated with water containing high levels of  $Mg^{2+}$  may produce low yields due to  $Mg^{2+}$ -induced  $Ca^{2+}$  deficiencies, but there is still insufficient data



TABLE 3

The total N and P in winery wastewater, river water and diluted winery wastewater used for the irrigation of Cabernet Sauvignon/99R during the 2010/2011, 2011/2012 and 2012/2013 seasons. The total N was not determined in the 2009/2010 season.

Treatment no. and target COD (mg/L)	Total N (mg/L)			H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> (mg/L)			
	2010/2011	2011/2012	2012/2013	2009/2010	2010/2011	2011/2012	2012/2013
Winery	4.96 ± 5.61	7.71 ± 8.67	16.45 ± 18.22	7.43 ± 8.37	22.11 ± 13.03	18.80 ± 8.63	10.82 ± 4.61
T1 - River water	1.34 ± 0.62	1.54 ± 1.01	1.95 ± 2.49	0.00 ± 0.00	0.02 ± 0.04	0.03 ± 0.03	0.24 ± 0.26
T2 - 100	0.65 ± 0.44	0.38 ± 0.33	0.59 ± 0.58	0.00 ± 0.00	0.02 ± 0.04	0.17 ± 0.19	0.23 ± 0.24
T3 - 250	0.47 ± 0.41	0.23 ± 0.30	0.28 ± 0.25	0.00 ± 0.00	0.18 ± 0.22	0.45 ± 0.39	0.28 ± 0.28
T4 - 500	0.24 ± 0.16	0.96 ± 0.88	0.34 ± 0.38	0.20 ± 0.35	0.64 ± 0.44	1.02 ± 0.92	0.54 ± 0.50
T5 - 1 000	0.38 ± 0.26	1.42 ± 1.88	0.76 ± 0.98	0.67 ± 1.15	1.64 ± 0.90	2.12 ± 1.74	1.12 ± 0.77
T6 - 1 500	0.80 ± 1.30	2.38 ± 3.50	1.363 ± 1.55	1.20 ± 1.82	2.65 ± 1.37	3.11 ± 2.44	1.53 ± 1.04
T7 - 2 000	0.98 ± 1.24	3.34 ± 4.69	2.16 ± 2.10	1.67 ± 2.39	3.87 ± 1.81	4.08 ± 3.91	2.08 ± 1.21
T8 - 2 500	1.00 ± 1.49	4.81 ± 5.72	3.13 ± 3.14	2.60 ± 2.76	4.85 ± 2.10	5.27 ± 4.91	2.55 ± 1.47
T9 - 3 000	1.28 ± 1.72	5.61 ± 7.66	2.68 ± 3.22	3.30 ± 3.40	6.26 ± 3.01	6.40 ± 5.92	3.04 ± 1.58

TABLE 4

The Ca<sup>2+</sup> and Mg<sup>2+</sup> in winery wastewater, river water and diluted winery wastewater used for irrigation of Cabernet Sauvignon/99R during the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	Ca <sup>2+</sup> (mg/L)				Mg <sup>2+</sup> (mg/L)			
	2009/10	2010/11	2011/12	2012/13	2009/10	2010/11	2011/12	2012/13
Winery	40.4 ± 20.4	24.9 ± 5.0	47.7 ± 17.3	29.3 ± 11.7	8.5 ± 1.9	9.8 ± 2.5	11.0 ± 4.9	9.9 ± 5.3
T1 - River water	5.4 ± 0.8	7.7 ± 0.6	5.2 ± 3.3	6.5 ± 2.4	3.5 ± 0.6	4.9 ± 0.2	3.4 ± 2.2	3.3 ± 1.5
T2 - 100	5.8 ± 0.7	7.8 ± 0.5	5.6 ± 3.1	7.2 ± 2.6	3.6 ± 0.6	5.0 ± 0.2	3.5 ± 2.1	3.6 ± 1.5
T3 - 250	6.3 ± 0.5	7.7 ± 0.9	6.8 ± 2.4	7.6 ± 2.6	3.7 ± 0.5	5.0 ± 0.2	3.8 ± 2.1	3.7 ± 1.5
T4 - 500	7.5 ± 1.0	8.8 ± 1.2	8.6 ± 0.8	8.5 ± 2.7	3.9 ± 0.5	5.2 ± 0.3	3.9 ± 1.9	4.0 ± 1.4
T5 - 1 000	10.0 ± 2.9	9.7 ± 1.5	11.8 ± 2.3	9.6 ± 2.6	4.2 ± 0.4	5.5 ± 0.4	4.4 ± 1.8	4.5 ± 1.5
T6 - 1 500	12.0 ± 4.7	10.9 ± 1.8	14.2 ± 4.0	10.0 ± 2.3	4.5 ± 0.3	5.7 ± 0.5	4.8 ± 1.9	4.6 ± 1.4
T7 - 2 000	13.9 ± 6.4	11.8 ± 2.0	17.8 ± 8.2	11.5 ± 3.0	4.7 ± 0.4	6.1 ± 0.6	5.1 ± 1.5	5.0 ± 1.5
T8 - 2 500	15.8 ± 6.5	12.4 ± 2.2	20.6 ± 11.2	12.6 ± 3.0	5.3 ± 0.1	6.2 ± 0.8	5.5 ± 1.3	5.5 ± 1.8
T9 - 3 000	17.5 ± 7.5	13.5 ± 2.5	24.1 ± 14.3	13.0 ± 2.8	5.3 ± 0.5	6.5 ± 0.9	6.0 ± 1.2	5.6 ± 1.8

to make the Ca<sup>2+</sup>-to-Mg<sup>2+</sup> ratio an evaluation factor (Ayers & Westcott, 1985). However, should this ratio be less than one, or the Ca<sup>2+</sup>-to-total-cation ratio less than 0.15, the potential negative effects of Na<sup>+</sup> may be exacerbated (Ayers & Westcott, 1985). If this is the case, a further evaluation of the water is required. Under the conditions of this study, the Ca<sup>2+</sup>-to-Mg<sup>2+</sup> ratio was consistently higher than one (data not shown). Therefore, it is expected that the high Ca<sup>2+</sup>-to-Mg<sup>2+</sup> ratios would have suppressed any possible negative effects of Na.

The K<sup>+</sup> concentrations in the diluted wastewater increased substantially with a decrease in dilution, *i.e.* an increase in the COD level of the water (Table 5). Mulidzi *et al.* (2009) tentatively considered 200 mg/L K<sup>+</sup> in winery wastewater as being high. In terms of this norm, the K<sup>+</sup> levels in the diluted wastewater were generally not high. Given that the K<sup>+</sup> concentrations in the diluted wastewater were still substantially lower than in the undiluted wastewater, dilution

had a positive effect on the K<sup>+</sup>. It should be borne in mind that the K<sup>+</sup> in the wastewaters could make an important contribution to the K<sup>+</sup> nutrient requirements of the grapevine. On the negative side, it must be noted that increasing the amount of K<sup>+</sup> may result in a decrease in the hydraulic conductivity and infiltration rate of soils (Levy & Van der Watt, 1990). There is a broad spectrum of possible effects of K<sup>+</sup> on infiltration, ranging from being similar to Na<sup>+</sup> to being similar to Ca<sup>2+</sup>. Furthermore, it was concluded that, relative to Ca<sub>ex</sub> and Na<sub>ex</sub>, K<sub>ex</sub> had an intermediate effect on the soil hydraulic properties (Arienzo *et al.*, 2009b). According to Laurenson *et al.* (2012), the PAR has been less widely adopted than the SAR, as K<sup>+</sup> is low in most wastewaters. However, the PAR has proved important for wastewaters of high K<sup>+</sup> concentration, *i.e.* piggery, meat-processing and winery wastewaters (Smiles & Smith, 2004). The PAR in the diluted wastewater increased with a decrease in the level of dilution (data not shown). All the values, with only one exception, were lower than the values of 2.1 to 3.2 reported

TABLE 5

The  $K^+$  and  $Na^+$  in winery wastewater, river water and diluted winery wastewater used for irrigation of Cabernet Sauvignon/99R during the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	$K^+$ (mg/L)				$Na^+$ (mg/L)			
	2009/2010	2010/2011	2011/2012	2012/2013	2009/2010	2010/2011	2011/2012	2012/2013
Winery	621 ± 265	242 ± 109	314 ± 93	189 ± 58	121 ± 50	93 ± 44	91 ± 11	86 ± 49
T1 - River water	3 ± 1	2 ± 1	2 ± 2	2 ± 1	10 ± 1	13 ± 1	12 ± 6	10 ± 3
T2 - 100	9 ± 5	4 ± 4	5 ± 4	4 ± 2	12 ± 1	14 ± 2	13 ± 6	11 ± 3
T3 - 250	18 ± 11	11 ± 10	11 ± 4	9 ± 4	13 ± 1	18 ± 6	15 ± 5	13 ± 3
T4 - 500	34 ± 13	21 ± 17	21 ± 9	16 ± 8	17 ± 1	25 ± 12	18 ± 3	15 ± 3
T5 - 1 000	74 ± 42	40 ± 33	38 ± 14	28 ± 12	24 ± 6	31 ± 20	24 ± 6	19 ± 4
T6 - 1 500	106 ± 60	55 ± 42	52 ± 20	35 ± 16	30 ± 8	38 ± 24	28 ± 11	21 ± 6
T7 - 2 000	135 ± 76	75 ± 59	63 ± 23	45 ± 16	35 ± 10	42 ± 26	35 ± 18	24 ± 7
T8 - 2 500	163 ± 89	89 ± 70	84 ± 38	56 ± 21	43 ± 9	46 ± 32	41 ± 25	28 ± 9
T9 - 3 000	198 ± 114	112 ± 95	100 ± 47	65 ± 26	49 ± 12	52 ± 38	46 ± 31	32 ± 11

for winery wastewater by Laurenson *et al.* (2012). These values were generally lower than the PAR values, ranging from 3.7 to 43, reported for a winery in Australia (Arienzo *et al.*, 2009b). It was previously shown that soil hydraulic conductivity was considerably reduced when the PAR exceeded 20 in a laboratory study (Arienzo *et al.*, 2009b). These negative effects occurred even when the electrolyte concentrations in the soil were relatively high, *i.e.* > 40 meq/L. However, it was also shown that the negative effect of  $Na^+$  was more pronounced compared to  $K^+$  at the same electrolyte concentration.

As the field work progressed, it became evident that the  $Na^+$  concentration in the river water used for dilution was consistently high. Sampling water at three places along the course of the Holsloot River during the 2012/2013 season (data not shown) revealed that the  $Na^+$  levels increased substantially along the floodplain during summer, up to the point where the water was abstracted near the Goudini winery. However, the  $Na^+$  concentrations in spring (September) were more comparable along the course of the river, probably due to the high flow that occurred in the winter. Since no definite point of contamination could be identified, the  $Na^+$  probably became more concentrated under low flow conditions. As expected,  $Na^+$  levels in the diluted wastewaters increased substantially with a decrease in dilution of the winery wastewater (Table 5). Grapevines are considered moderately sensitive to foliar injury from  $Na^+$ . Therefore, a concentration of 115 mg/L  $Na^+$  in the water is recommended as the upper threshold for overhead irrigation (Department of Water Affairs & Forestry, 1996). As the experiment vineyard was irrigated by means of micro-sprinklers, the leaves were not expected to be wetted during irrigation. However, the  $Na^+$  levels in the diluted wastewater treatments were so low that, even if the leaves were wetted, no substantial damage would be expected. The abovementioned  $Na^+$  threshold was exceeded only on one occasion during the 2010/2011 season, when the  $Na^+$  concentration amounted to 124 mg/L in winery wastewater diluted to a COD level of 3 000 mg/L (data not shown). In general, the levels of  $Na^+$  in the undiluted wastewater also did not exceed the threshold (Table 5).

As expected, the SAR increased with a decrease in the level of dilution of the wastewater (Table 6). Since the  $Ca^{2+}$  and  $Mg^{2+}$  also increased, this suggests that the increase in these cations could not counterbalance the effect of increased  $Na^+$  on the SAR. The SAR norm stipulated by the General Authorisations of 2013 (Department of Water Affairs, 2013) was only exceeded on 2011-04-14, when the SAR of the irrigation waters diluted to COD levels of 1 500 mg and higher was more than 5 (data not shown). Furthermore, the SAR in the diluted winery wastewaters was still within acceptable limits for grapevine irrigation, *i.e.* less than *c.* 10 (Richards, 1954; Myburgh, 2012). The foregoing shows that the diluted winery wastewater would pose no sodicity hazard if used for irrigation under the given conditions. It must be noted that the SAR in the undiluted winery wastewater also did not exceed the acceptable threshold for grapevine irrigation (data not shown).

Similar to  $Na^+$ , it became evident that the  $Cl^-$  concentrations in the river water used for dilution were consistently high. Sampling water along the course of the Holsloot River during the 2012/2013 season revealed that the  $Cl^-$  levels increased substantially during summer along the floodplain up to the point where water was abstracted near the winery (data not shown). However, the  $Cl^-$  concentrations in spring were more comparable along the course of the river, probably due to high flow in the winter. Since no definite point of contamination could be identified, the  $Cl^-$  probably became more concentrated under low flow conditions. Although the  $Cl^-$  concentrations in the diluted waters were lower than that of the undiluted winery wastewater, the range of dilutions did not cause any consistent trends with respect to the level of dilution (Table 7). Levels in both the diluted and undiluted winery wastewater were substantially lower than 150 mg/L and 700 mg/L, which are the threshold for grapevine root uptake and overhead irrigation respectively (Van Zyl, 1981). This suggests that  $Cl^-$  levels in the diluted wastewater would not have caused damage to the grapevines, even if the leaves were wetted. Although the  $HCO_3^-$  concentration increased with a decrease in the dilution of the wastewater, the concentrations varied considerably between seasons,

*i.e.* high standard deviation values (Table 7). Similarly, the  $\text{HCO}_3^-$  concentration also showed large variation within a season. The reason for the variability is uncertain. However, the concentration in the least diluted water (T9) was still substantially lower compared to the undiluted winery wastewater. When irrigations were applied on 2011-02-09, 2011-03-30, 2012-03-06 and 2012-03-19, no trends with regard to the level of dilution were evident for  $\text{HCO}_3^-$  concentrations in the water (data not shown). Throughout the 2012/2013 season, no trends were evident for  $\text{HCO}_3^-$  levels in the water, with the exception of on 2013-04-30 (data not shown). At this stage, there is no explanation why the  $\text{HCO}_3^-$  level did not increase with a decrease in the level of dilution on these days.

Irrigation water containing high levels of  $\text{HCO}_3^-$  can negatively affect plants, soils and irrigation systems. Irrigation waters that contain high levels of  $\text{HCO}_3^-$  and  $\text{CO}_3^{2-}$  can increase  $\text{HCO}_3^-$  in the soil solution. Consequently,  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  can precipitate as insoluble carbonates when the soil dries out (Van Zyl, 1981; McCarthy *et al.*, 1988). According to the norms proposed for  $\text{HCO}_3^-$  concentrations

in overhead irrigation water, values lower than 91.5 mg/L (1.5 me/L) indicate no restriction when used, whereas levels between 91.5 mg/L and 518.6 mg/L (8.5 me/L) indicate a slight to moderate degree of restriction when used (Ayers & Westcott, 1985). In the first season, dilutions above 500 mg/L COD contained excessive levels of  $\text{HCO}_3^-$ , whereas in the second and third seasons, dilutions above 1 000 mg/L COD contained excessive  $\text{HCO}_3^-$  (data not shown). In contrast, the  $\text{HCO}_3^-$  levels fell into the no restriction category during the 2012/2013 season. There are no recently recommended guidelines (Department of Water Affairs & Forestry, 1996; ANZECC, 2000).

Although the  $\text{SO}_4^{2-}$  in the diluted irrigation waters generally increased with a decrease in the level of dilution of the wastewater, the levels in the least diluted water (T9) were substantially lower compared to those in the winery wastewater (Table 7). Similar to  $\text{HCO}_3^-$ , the  $\text{SO}_4^{2-}$  concentrations varied considerably between seasons (Table 7), and within a season (data not shown). The reason for the variability is also uncertain. In general,  $\text{SO}_4^{2-}$  levels in the diluted winery wastewater were below the proposed

TABLE 6

The sodium adsorption ratio (SAR) in winery wastewater, river water and diluted winery wastewater used for irrigation of Cabernet Sauvignon/99R during the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	SAR			
	2009/2010	2010/2011	2011/2012	2012/2013
Winery	4.6 ± 1.1	4.3 ± 2.2	3.2 ± 0.7	3.4 ± 1.5
T1 - River water	0.9 ± 0.0	0.9 ± 0.2	1.0 ± 0.2	0.8 ± 0.2
T2 - 100	1.0 ± 0.1	0.9 ± 0.3	1.1 ± 0.2	0.8 ± 0.1
T3 - 250	1.1 ± 0.1	1.2 ± 0.5	1.2 ± 0.2	0.9 ± 0.2
T4 - 500	1.3 ± 0.1	1.4 ± 0.8	1.3 ± 0.2	1.1 ± 0.2
T5 - 1 000	1.7 ± 0.3	1.8 ± 1.2	1.5 ± 0.4	1.3 ± 0.3
T6 - 1 500	1.9 ± 0.3	2.2 ± 1.5	1.6 ± 0.6	1.4 ± 0.4
T7 - 2 000	2.1 ± 0.3	2.4 ± 1.4	1.8 ± 0.7	1.5 ± 0.4
T8 - 2 500	2.5 ± 0.2	2.5 ± 1.8	2.0 ± 0.9	1.7 ± 0.5
T9 - 3 000	2.7 ± 0.3	2.9 ± 1.9	2.1 ± 1.0	1.8 ± 0.6

TABLE 7

The  $\text{Cl}^-$ ,  $\text{HCO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{B}^{3+}$  and  $\text{Fe}^{2+}$  in winery wastewater, river water and diluted winery wastewater used for irrigation of Cabernet Sauvignon/99R. Data are means for the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	$\text{Cl}^-$	$\text{HCO}_3^-$	$\text{SO}_4^{2-}$	$\text{B}^{3+}$	$\text{Fe}^{2+}$
	(mg/L)				
Winery	50.6 ± 13.6	537 ± 520	138 ± 67	0.80 ± 0.31	3.81 ± 1.50
T1 - River water	26.3 ± 3.0	13 ± 6	23 ± 3	0.03 ± 0.02	0.12 ± 0.14
T2 - 100	25.9 ± 3.0	19 ± 11	21 ± 4	0.02 ± 0.01	0.24 ± 0.29
T3 - 250	25.6 ± 3.7	30 ± 19	21 ± 5	0.04 ± 0.01	0.23 ± 0.18
T4 - 500	25.4 ± 4.1	47 ± 34	25 ± 6	0.06 ± 0.03	0.31 ± 0.16
T5 - 1 000	26.2 ± 3.6	77 ± 64	32 ± 10	0.12 ± 0.06	0.37 ± 0.13
T6 - 1 500	27.9 ± 3.7	109 ± 84	34 ± 8	0.16 ± 0.09	0.60 ± 0.20
T7 - 2 000	27.0 ± 4.7	142 ± 115	51 ± 39	0.22 ± 0.12	0.74 ± 0.27
T8 - 2 500	27.8 ± 4.8	171 ± 146	75 ± 52	0.28 ± 0.16	0.97 ± 0.41
T9 - 3 000	31.4 ± 6.0	212 ± 176	75 ± 54	0.34 ± 0.19	1.22 ± 0.53

level of 150 mg/L and lower for reclaimed effluent water quality standards for vineyard re-use (Ryder, 1995). In the 2009/2010 and 2011/2012 seasons,  $\text{SO}_4^{2-}$  levels in the undiluted wastewater were above this optimum level, but still lower than the maximum threshold of 250 mg/L.

The  $\text{B}^{3+}$  levels in the diluted wastewater increased with a decrease in level of dilution (Table 7). Concentrations in the least diluted water were still substantially lower than those in the undiluted winery wastewater. Although  $\text{B}^{3+}$  is essential for the growth of plants, it reaches toxic levels at low concentrations. Grapevines have been classed as sensitive (Ayers & Westcott, 1985; Department of Water Affairs & Forestry, 1996; ANZECC, 2000) to highly sensitive (Van Zyl, 1981) with regard to B toxicity. In general,  $\text{B}^{3+}$  levels of 0.5 mg/L are considered ideal for vineyard irrigation (McCarthy *et al.*, 1988), whereas levels under 0.75 mg/L have been recommended by Ayers and Westcott (1985). On 2011-04-13, the limit of 0.5 mg/L was only exceeded where winery wastewater was diluted to 3 000 mg/L (T9), whereas on 2011-04-14, winery wastewater diluted to 1 500 mg/L and more (T6 to T9) exceeded this limit (data not shown). It should be noted that, due to low COD levels in the winery wastewater in April 2011, the range of COD levels had to be made up and applied on two consecutive days. On 2012-05-02, diluted winery wastewater of 1 000 mg/L and higher (T5 to T9) also exceeded the limit of 0.5 mg/L. The foregoing shows that diluted winery wastewater has a sporadic risk of inducing  $\text{B}^{3+}$  toxicity if used for vineyard irrigation. The  $\text{Fe}^{2+}$  levels increased with a decrease in the dilution of the wastewater, but the level in the least diluted wastewater was still substantially lower compared to the undiluted winery wastewater (Table 7). Recommended maximum levels of  $\text{Fe}^{2+}$  in irrigation water for continuous irrigation on all soils is 5 mg/L (Van Zyl, 1981). With the exception of the 2011/2012 season,  $\text{Fe}^{2+}$  levels in the undiluted wastewaters never exceeded this value. However, the Fe concentration becomes important in the case of drip irrigation, where major clogging problems can be expected when  $\text{Fe}^{2+}$  levels are higher than 1.5 mg/L (Department of Water Affairs & Forestry, 1996). Given the low  $\text{Fe}^{2+}$  concentrations in the

diluted wastewater, it would not cause clogging of micro-sprinkler systems under the prevailing conditions.

#### Amount of elements applied

In terms of total N, amounts added *via* the irrigation water were generally higher for T9 compared to the river water control (T1), but trends across the range of COD levels were inconsistent (Table 8). The total N applied *via* the winery wastewater diluted to 2 000 mg/L COD and higher was similar to the estimated c. 5 kg N/ha applied *via* winery wastewater based on an irrigation depth of 100 mm (Laurenson *et al.*, 2012). Full-bearing grapevines annually require 50 kg N if 10 to 15 tons of fruit is produced per ha (Conradie, 1994). Based on this recommendation, the amount of N applied *via* the diluted wastewater appeared to be completely inadequate to supply the grapevine's annual N requirement under the prevailing conditions (Fig. 1A). Therefore, winery wastewater cannot be considered as a source of N. The amount of  $\text{H}_2\text{PO}_4^-$  applied *via* the irrigation water increased with a decrease in wastewater dilution (Table 8). The  $\text{H}_2\text{PO}_4^-$  applied *via* the winery wastewater diluted to 2 000 mg/L COD and higher was similar to the estimated c. 5.3 kg P per ha applied *via* winery wastewater based on an irrigation depth of 100 mm (Laurenson *et al.*, 2012). Full-bearing grapevines annually require 0.7 kg P per ton of fruit produced (Conradie, 1994). Based on this recommendation, the amount of P applied *via* the winery wastewater diluted to 2 500 mg/L COD and higher would supply adequate P during most seasons if the grape yield amounted to 10 t/ha under the prevailing conditions (Fig. 1B).

The amount of  $\text{K}^+$  applied per hectare increased substantially with a decrease in dilution of the winery wastewater (Table 8). In 2010/2011 and 2012/2013, similar amounts were applied in the pre- and post-harvest periods (data not shown). Since only one irrigation was applied in the post-harvest period of 2011/2012, amounts of  $\text{K}^+$  added *via* the irrigation water were substantially less than during the pre-harvest period (data not shown). In general,  $\text{K}^+$  applied *via* winery wastewater diluted to 2 500 mg/L and higher was more than the estimated 129 kg  $\text{K}^+$  per hectare applied *via* winery wastewater based on an irrigation depth of 100 mm

TABLE 8

The calculated total amounts of total N,  $\text{H}_2\text{PO}_4^-$  and  $\text{K}^+$  applied *via* river water and diluted winery wastewater used for irrigation of Cabernet Sauvignon/99R during the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	Total-N (kg/ha)			$\text{H}_2\text{PO}_4^-$ (kg/ha)				$\text{K}^+$ (kg/ha)			
	2010/ 2011	2011/ 2012	2012/ 2013	2009/ 2010 <sup>(1)</sup>	2010/ 2011	2011/ 2012	2012/ 2013	2009/ 2010	2010/ 2011	2011/ 2012	2012/ 2013
T1 - River water	5.30	3.32	6.45	0.00	0.09	0.07	0.70	4.93	7.46	6.56	5.71
T2 - 100	3.61	1.46	3.08	0.00	0.08	0.22	0.66	12.89	13.52	11.01	9.84
T3 - 250	3.28	1.25	2.16	0.00	0.39	0.64	0.81	23.42	25.14	21.58	22.17
T4 - 500	2.62	2.03	2.42	0.25	1.19	1.37	1.42	43.10	43.86	35.86	41.05
T5 - 1 000	3.01	2.21	3.50	0.81	3.18	2.96	2.86	92.84	79.04	65.13	70.65
T6 - 1 500	4.30	3.09	4.97	1.49	5.35	4.43	3.94	132.86	112.83	87.32	88.26
T7 - 2 000	4.76	4.25	6.95	2.04	8.46	5.24	5.22	166.97	148.79	103.95	110.53
T8 - 2 500	4.69	6.31	9.22	3.18	10.38	6.83	6.24	200.08	173.41	134.46	135.37
T9 - 3 000	5.42	6.41	8.20	4.04	13.46	8.02	7.52	243.63	214.84	157.67	159.50

<sup>(1)</sup> In 2009/2010, irrigations were only applied in the post-harvest period.



(Laurenson *et al.*, 2012). Full-bearing grapevines annually require 3 kg K<sup>+</sup> per ton of fruit produced (Conradie, 1994). Based on this recommendation, the amount of K<sup>+</sup> applied *via* winery wastewater diluted to 250 mg/L COD and higher would

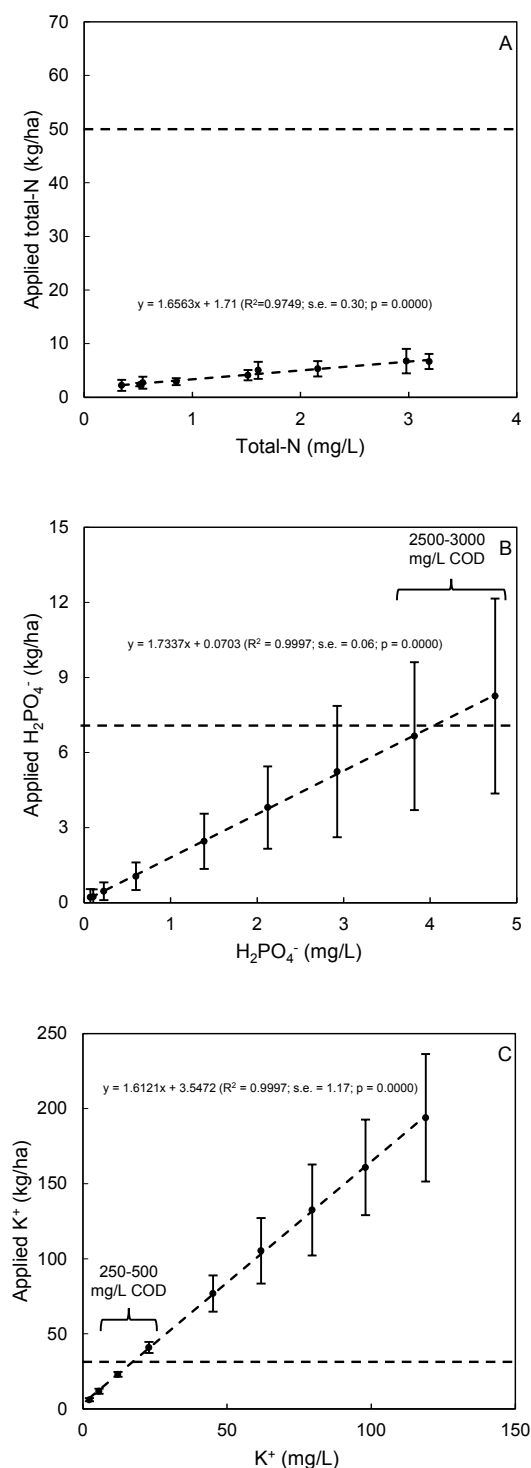


FIGURE 1

Relationship between amounts of (A) total N, (B) H<sub>2</sub>PO<sub>4</sub><sup>-</sup> and (C) K<sup>+</sup> applied *via* irrigation with diluted winery wastewater and the total N, H<sub>2</sub>PO<sub>4</sub><sup>-</sup> and K<sup>+</sup> concentration in the water. Dashed horizontal lines indicate the annual grapevine requirement for a grape yield of 10 t/ha according to Conradie (1981, 1994). With the exception of total N, data are means for four seasons. Vertical bars indicate standard deviation.

supply more than adequate K<sup>+</sup> if the grape yield amounted to 10 t/ha under the prevailing conditions (Fig. 1C). As the grapevine's nutrient requirements are generally low during the harvest and post-harvest periods (Conradie, 1981), the K<sup>+</sup> supplied *via* the wastewater will only be beneficial if it is not leached from the root zone during winter. On average, between 47 kg/ha and 164 kg/ha K<sup>+</sup> was applied in excess to the soil where the winery wastewater was diluted to COD levels ranging from 1 000 mg/L (T5) to 3 000 mg/L (T9). The effect of high concentrations of K<sup>+</sup> applied to soils, as well as the fate of K in soils and grapevines irrigated with winery wastewater, has received limited attention (Mosse *et al.*, 2011; Laurenson *et al.*, 2012). However, excessive K<sup>+</sup> applied *via* the diluted winery wastewater could have several implications. Excessive K<sup>+</sup> in grape berries can be detrimental to wine quality, as it decreases free tartaric acid (Mpelasoka *et al.*, 2003). Subsequently, the pH in grape juice, must and wine will increase (Saayman, 1981; Mpelasoka *et al.*, 2003). Excessive K<sup>+</sup> in fruit also causes the formation of insoluble potassium bitartrate (Laurenson *et al.*, 2012).

The increase in pH causes unstable musts and wines, as well as a reduction in the degree of ionisation of anthocyanins (Mpelasoka *et al.*, 2003). The increase in berry pH in hot climates produces grape juice with a high pH, which has a flat taste and possible brown hue (Kodur, 2011; Laurenson *et al.*, 2012). In addition to these grapevine responses, excessive K<sup>+</sup> can reduce juice N (Saayman, 1981), thereby increasing the risk of stuck fermentation during the winemaking process. Excessive K<sup>+</sup> can also reduce Ca<sup>2+</sup> and Mg<sup>2+</sup> in the grapevine, indicating antagonisms between K<sup>+</sup> and these elements (Morris & Cawthon, 1982; Wolf *et al.*, 1983; Myburgh & Howell, 2014). Given that the amounts of K<sup>+</sup> applied *via* the diluted winery wastewater were much higher than the requirements of the grapevine, the cultivation of an interception crop during summer might be useful to absorb excessive K<sup>+</sup>.

The amounts of Ca<sup>2+</sup> and Mg<sup>2+</sup> applied increased with a decrease in dilution of the winery wastewater (Table 9), but differences between the highest level of dilution, *i.e.* T2, and the lowest level of dilution, *i.e.* T9, were not as substantial as in the case of K<sup>+</sup> and Na<sup>+</sup> (Tables 8 & 9). Full-bearing grapevines require *c.* 2 kg Ca<sup>2+</sup> annually per ton of fruit produced (Conradie, 1981). Based on this recommendation, the amount of Ca<sup>2+</sup> applied *via* winery wastewater diluted to 500 mg/L COD and higher would supply more than adequate Ca<sup>2+</sup> if the grape yield amounted to 10 t/ha under the prevailing conditions (Fig. 2A). As the grapevine's nutrient requirements are generally low during the harvest and post-harvest periods (Conradie, 1981), the Ca<sup>2+</sup> supplied *via* the wastewater will only be beneficial if it is not leached from the root zone during winter. Furthermore, bunches require no Ca<sup>2+</sup> during véraison and harvest. With regard to Mg<sup>2+</sup>, 0.7 kg is required per ton of grapes produced. Under the prevailing conditions, all of the treatments supplied sufficient Mg<sup>2+</sup> to supply the grapevine's requirements (Fig. 2B). The amounts of Na<sup>+</sup> applied per hectare increased substantially with a decrease in level of dilution of the winery wastewater (Table 9). In 2010/2011 and 2012/2013, similar amounts were applied in the pre- and post-harvest

periods (data not shown). As there was only one irrigation in the 2011/2012 post-harvest period, the amounts of  $\text{Na}^+$  added *via* the irrigation water were substantially less than during the pre-harvest period (data not shown). The total amounts of

$\text{Na}^+$  added *via* the irrigation water ranged from 32 kg/ha for the river water control (T1) to 85 kg/ha for the least diluted water, *viz.* 3 000 mg/L COD (Fig. 2C). Although there are no threshold values for grapevines with regard to the amount of  $\text{Na}^+$  applied per hectare, it is well known that excessive

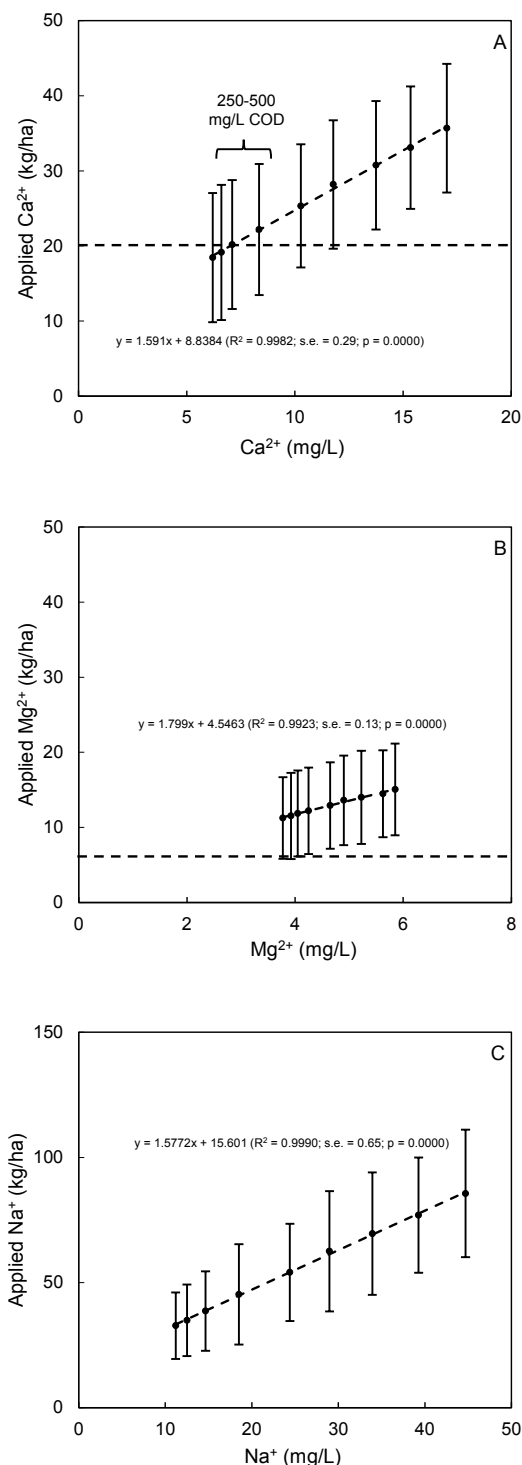


FIGURE 2

Relationship between amounts of (A)  $\text{Ca}^{2+}$ , (B)  $\text{Mg}^{2+}$  and (C)  $\text{Na}^+$  applied *via* irrigation with diluted winery wastewater and the  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{Na}^+$  concentration in the water. Dashed horizontal lines indicate the annual grapevine requirement for a grape yield of 10 t/ha, according to Conradie (1981, 1994). Data are means for four seasons. Vertical bars indicate standard deviation.

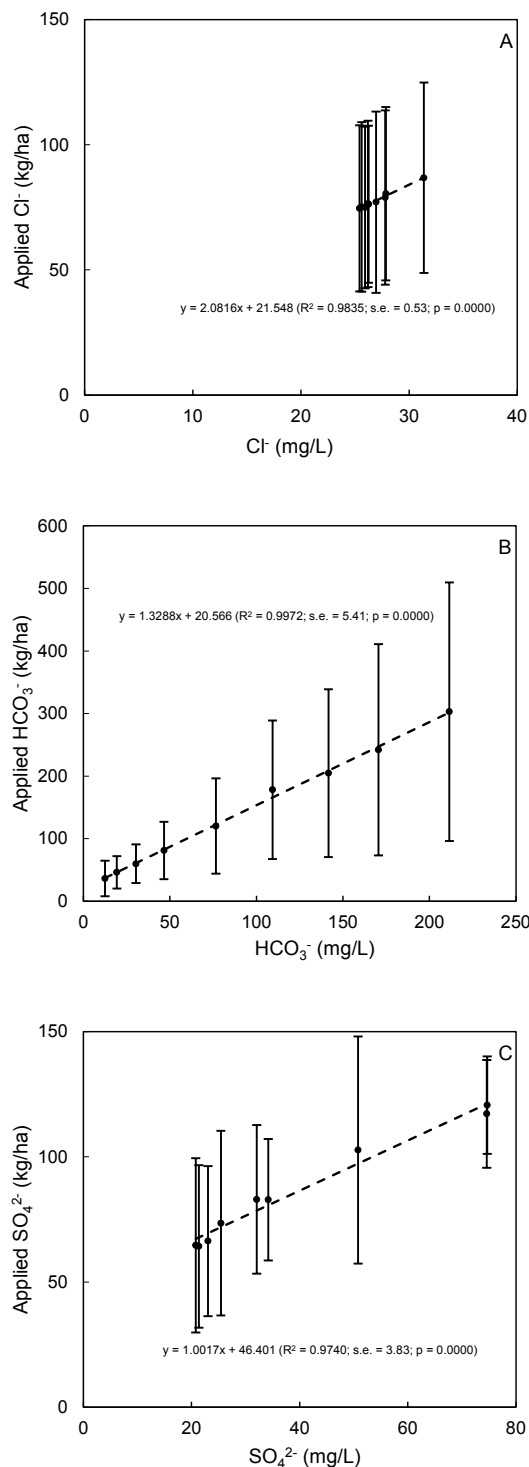


FIGURE 3

Relationship between amounts of (A)  $\text{Cl}^-$ , (B)  $\text{HCO}_3^-$  and (C)  $\text{SO}_4^{2-}$  applied *via* irrigation with diluted winery wastewater and the  $\text{Cl}^-$ ,  $\text{HCO}_3^-$  and  $\text{SO}_4^{2-}$  concentration in the water. Data are means for four seasons. Vertical bars indicate standard deviation.

TABLE 9

The calculated total amounts of  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{Na}^{+}$  applied *via* river water and diluted winery wastewater used for irrigation of Cabernet Sauvignon/99R during the 2009/2010, 2010/2011, 2011/2012 and 2012/2013 seasons.

Treatment no. and target COD (mg/L)	$\text{Ca}^{2+}$ (kg/ha)				$\text{Mg}^{2+}$ (kg/ha)				$\text{Na}^{+}$ (kg/ha)			
	2009/ 2010 <sup>(1)</sup>	2010/ 2011	2011/ 2012	2012/ 2013	2009/ 2010	2010/ 2011	2011/ 2012	2012/ 2013	2009/ 2010	2010/ 2011	2011/ 2012	2012/ 2013
T1 - River water	9.76	30.02	14.94	19.07	6.44	19.08	9.66	9.82	18.35	50.36	32.80	29.52
T2 - 100	9.55	30.74	15.43	20.84	6.05	19.59	9.86	10.61	19.04	53.59	35.50	31.45
T3 - 250	10.29	30.79	17.51	22.11	6.22	19.83	10.40	11.02	21.36	59.83	37.53	35.81
T4 - 500	11.87	32.90	20.05	23.89	6.55	20.21	10.57	11.53	25.24	73.05	42.42	40.29
T5 - 1000	14.91	34.92	24.95	26.53	6.92	20.71	11.34	12.70	34.99	81.17	49.46	50.71
T6 - 1500	17.58	38.51	28.57	28.09	7.31	21.64	12.19	13.27	41.90	97.30	54.90	55.96
T7 - 2000	19.30	39.94	32.49	31.22	7.28	22.23	12.44	14.09	47.83	104.65	61.45	64.24
T8 - 2500	21.74	40.79	36.45	33.35	8.11	22.05	12.82	14.92	56.85	110.12	69.07	71.69
T9 - 3000	23.89	43.13	40.69	35.02	8.13	22.92	13.67	15.51	64.51	122.33	73.97	81.57

<sup>(1)</sup> In 2009/2010, irrigations were only applied in the post-harvest period.

$\text{Na}^{+}$  can reduce vegetative growth, yield and suppress  $\text{Ca}^{2+}$  uptake (Myburgh & Howell, 2014 and references therein).

Although the amounts of  $\text{Cl}^{-}$  added *via* the irrigation water were higher for T9 compared to the river water control (T1), increases across the COD levels were inconsistent (data not shown). It should be noted that the river water also contained relatively high levels of  $\text{Cl}^{-}$  (Table 5). The total amounts of  $\text{Cl}^{-}$  added *via* the irrigation water ranged from 76 kg/ha for the river water control to 87 kg/ha for the least diluted water, *viz.* 3 000 mg/L COD (Fig. 3A). Although there are no threshold values for grapevines with regard to amount of  $\text{Cl}^{-}$  applied per hectare, excessive  $\text{Cl}^{-}$  can reduce vegetative growth and yield (Myburgh & Howell, 2014 and references therein). In 2010/2011 and 2011/2012 there was a substantial increase in  $\text{HCO}_3^{-}$  applied per ha with a decrease in dilution of the wastewater (data not shown). The total amounts of  $\text{HCO}_3^{-}$  added *via* the irrigation water ranged from 36 kg/ha for the river water control to 303 kg/ha for the least diluted water, *viz.* 3 000 mg/L COD (Fig. 3B). In general, the amount of  $\text{SO}_4^{2-}$  applied increased with increasing level of COD (data not shown). The total amounts of  $\text{SO}_4^{2-}$  added *via* the irrigation water ranged from 66 kg/ha for the river water control to 117 kg/ha for the least diluted water, *viz.* 3 000 mg/L COD (Fig. 3C).

Despite low levels of  $\text{B}^{3+}$  being applied *via* the irrigation water, amounts increased with a decrease in level of dilution of the winery wastewater (data not shown). According to Conradie (1994), the requirements of the grapevine for  $\text{B}^{3+}$  are relatively low. Under the prevailing conditions, less than 1 kg of  $\text{B}^{3+}$  was applied *via* the diluted winery wastewater. The amounts of  $\text{Fe}^{2+}$  applied per hectare increased with a decrease in the level of dilution of the winery wastewater (data not shown). According to Conradie (1994), the requirements of the grapevine for  $\text{Fe}^{2+}$  are relatively low. Under the prevailing conditions, *c.* 2 kg of  $\text{Fe}^{2+}$  per ha was applied *via* the diluted winery wastewater. The concentrations of  $\text{Cd}^{2+}$ ,  $\text{Cr}^{2+}$  and  $\text{As}^{3-}$  in the undiluted winery wastewater were extremely low, therefore amounts applied *via* the diluted winery wastewater

were low (data not shown). Furthermore, the heavy metals did not show any trends with regard to level of dilution.

## CONCLUSIONS

In previous studies, artificial “winery wastewater” was used. Furthermore, most of these studies were carried out in laboratories. This study was the first in which wastewater from a commercial winery was diluted with raw river water to a range of COD levels for vineyard irrigation at field scale. Since the pH in the diluted wastewater was lower than 6, it could induce nutrient toxicity if used for the irrigation of vineyards or other crops. The results indicate that the dilution of winery wastewater did not have any positive effect on pH with respect to irrigation water quality. The diluted winery wastewater did not pose any salinity hazard, since  $\text{EC}_{\text{iw}}$  was well below 2 dS/m. As the EC in the undiluted water was close to 2 dS/m, the dilution of winery wastewater reduced the salinity hazard with respect to irrigation water quality. Given the fact that the COD in the undiluted winery water generally was not lower than 7 624 mg/L, the dilution of winery wastewater had a positive effect on the COD in terms of irrigation water quality. The  $\text{K}^{+}$  concentrations in the diluted wastewater were substantially lower than in the undiluted wastewater. The  $\text{K}^{+}$  levels in the diluted wastewater were generally not high for winery wastewater, *i.e.* less than 200 mg/L. It must be noted that the  $\text{K}^{+}$  in the wastewater could make a contribution to the  $\text{K}^{+}$  requirements of the grapevine, *i.e.* if it was not lost *via* leaching during winter. For the given range of wastewater dilutions, the SAR never exceeded 10, which indicates that the diluted wastewater posed no sodicity hazard. Sodium and  $\text{Cl}^{-}$  never exceeded 115 and 150 mg/L, *i.e.* the respective upper toxicity thresholds of these elements for grapevines.

Considering the classical water quality criteria, *viz.* pH, EC and SAR, the dilution of winery wastewater up to a COD level of 3 000 mg/L produced irrigation water of which the quality would permit sustainable vineyard irrigation under the prevailing conditions, *viz.* a Mediterranean climate with high winter rainfall and sandy soil. As expected, the levels of  $\text{H}_2\text{PO}_4^{-}$ ,  $\text{K}^{+}$ ,  $\text{Na}^{+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{HCO}_3^{-}$ ,  $\text{SO}_4^{2-}$  and  $\text{B}^{3+}$  in the

diluted wastewater increased with a decrease in the level of dilution. In contrast, levels of N and Cl<sup>-</sup> were inconsistent with regard to the level of dilution. The B<sup>3+</sup> concentrations in the undiluted winery wastewater indicated a potential risk of inducing B toxicity if used for vineyard irrigation. Since B<sup>3+</sup> levels in the least diluted wastewater exceeded the recommended norm sporadically, there is still a slight risk of B<sup>3+</sup> toxicity which should not be ignored. The results indicated that SO<sub>4</sub><sup>2-</sup> levels in the diluted winery wastewater were below the proposed level of 150 mg/L for effluent water quality standards for vineyard re-use. Since one of the incentives for diluting winery wastewater is that it could serve as a possible nutrient source, it is important to note that the N load in the diluted winery wastewater was completely inadequate to supply the grapevine's annual requirement. On the positive side, P loads in the winery wastewater diluted to 2 500 mg/L COD and higher could supply more than adequate P if the grape yield amounted to 10 t/ha.

Likewise, the dilution of winery wastewater to 250 mg/L COD and higher could supply more than adequate K<sup>+</sup> if grape yield amounted to 10 t/ha. However, the excessive K<sup>+</sup> applied *via* the diluted wastewater could increase juice pH, which could cause unstable musts and wines, as well as a reduction in the degree of ionisation of anthocyanins in the wine. Furthermore, excessive K<sup>+</sup> application could induce nutrient imbalances in the grapevine tissues, particularly antagonisms with respect to N, Ca<sup>2+</sup> and Mg<sup>2+</sup>. Given that the amounts of K<sup>+</sup> applied *via* the diluted winery wastewater were considerably higher than the grapevine's requirements, the cultivation and removal of a suitable interception crop during summer might be useful to absorb excessive K<sup>+</sup>. Since grapevine nutrient requirements are generally low during the harvest and post-harvest periods, the N, P and K<sup>+</sup> supplied *via* the wastewater will only be beneficial if they are not leached from the root zone during winter. Soil, grapevine and wine responses to irrigation with diluted winery wastewater will be presented and discussed in subsequent articles.

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